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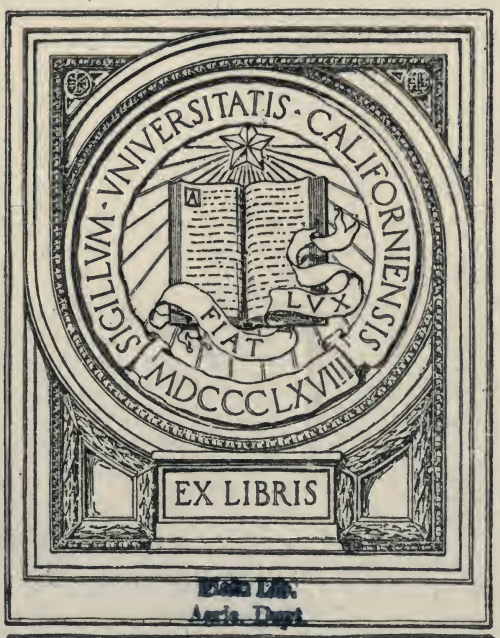
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UNIVERSITY OF CALIFORNIA

United States Department of Agriculture,

BUREAU OF CHEMISTRY—Circular No. 10 (Revised).

H. W. WILEY, Chief of Bureau.

METHODS OF ANALYSIS OF INSECTICIDES AND FUNGICIDES.

GENERAL DISCUSSION.

Samples of paris green, london purple, soft soap, copper carbonate and tobacco extracts are to be thoroughly mixed before analysis, taking care that the first two samples are not further pulverized. Samples of lye and potassium cyanid are to be weighed in large quantities in weighing bottles and aliquot portions from a water solution taken for analysis.

PARIS GREEN.

MOISTURE (PROVISIONAL).[1]

Dry 1 to 2 grams for 8 to 10 hours at 105° to 110° C, and calculate the loss as moisture.

TOTAL ARSENIOUS OXID, METHOD I (OFFICIAL).

SOLUTIONS REQUIRED.

A starch solution is used which is prepared by boiling 2 grams of starch with 200 cc of distilled water for about 5 minutes.

A standard iodin solution is prepared in the following manner: Dissolve 12.7 grams of powdered iodin in about 250 cc of water to which has been added 18 to 25 grams of C. P. potassium iodid, and make up the whole to a volume of 2 liters. To standardize this solution, weigh out 1 gram of C. P. dry arsenious oxid, transfer to a 250-cc flask by means of about 100 cc of a solution containing 2 grams of sodium hydroxid in each 100 cc, and boil until all arsenious oxid goes into solution. Make up to a volume of 250 cc and use 50 cc for analysis. This 50 cc portion is concentrated, by boiling in a 250-cc flask to half its volume, and allowed to cool to about 80° C. An equal volume of concentrated hydrochloric acid is now added, accompanied by 3 grams of potassium iodid, mixed, and the whole allowed to stand for 10 minutes (to reduce the arsenic oxid formed by boiling and the alkaline arsenite to arsenious oxid). The brown solution is then diluted with cold water and an approximately tenth-normal solution of sodium thiosulphate added drop by drop, until the solution becomes exactly colorless. (This end point is easy to read without the aid of starch.) This solution is made slightly alkaline with dry sodium carbonate (using a drop of methyl orange to read the change) and then is made slightly acid with hydrochloric acid, taking care that all lumps of sodium carbonate on the bottom are acted on by the hydrochloric acid. Sodium bicarbonate is now added in excess and the solution of iodin run in drop by drop, using starch water to read the end reaction. (Sometimes the solution gets dark toward the end of the titration. This change must not be confused with the final dark blue color given by the iodin and starch.)

[1] Terms "provisional" and "official" refer to adoption of methods by the Association of Official Agricultural Chemists.

From the number of cubic centimeters of iodin solution used and the weight of arsenious oxid taken, the value of each cubic centimeter of iodin in terms of arsenious oxid can be determined.

METHOD.

Two grams of paris green are weighed out and transferred to a 250-cc flask and about 100 cc of water and 2 grams of sodium hydroxid added. This mixture is boiled for 5 to 10 minutes, or until all the green particles have changed to red cuprous oxid. It is then cooled to room temperature and the volume made up to 250 cc. The well-shaken liquid is filtered through a dry filter and 50-cc portions taken for analysis. The analysis is carried out from this point forward the same as when standardizing the iodin solution.

Total Arsenious Oxid, Method II (Provisional).[1]

SOLUTIONS REQUIRED.

A standard, approximately twentieth-normal, solution of iodin is made as follows: Dissolve 12.7 grams of powdered iodin in 250 cc of water to which has been added 25 grams of potassium iodid, and make up to 2,000 cc. To standardize this solution weigh out 1 gram of dry C. P. arsenious oxid, transfer to a 250-cc flask, and dissolve by boiling about 20 minutes with 5 grams of sodium bicarbonate and about 100 cc of water; cool; add hydrochloric acid till acid, and sodium bicarbonate again until alkaline; make to the mark and take aliquots of 50 cc for standardizing.

A solution of sodium acetate containing 12.5 grams of the crystallized salt in each 25 cc.

A solution containing 2 to 3 grams of sodium potassium tartrate dissolved in each 50 cc.

A starch solution.

METHOD.

A gram of paris green is weighed out and transferred to a 100-cc flask, where it is boiled for 5 minutes with 25 cc of a solution of sodium acetate containing 12.5 grams of the crystallized salt. It is then made to the mark, shaken, and passed through a dry asbestos gooch filter. An aliquot portion of this filtrate is used to determine the soluble arsenious oxid. The residue on the filter is transferred to a beaker, and, after being beaten up with a little water, dissolved in hydrochloric acid by adding the concentrated acid a drop at a time. The whole is now transferred to the 100-cc flask and aliquot portions of 20 to 40 cc each taken for analysis. To each of these aliquots is added concentrated sodium carbonate solution a drop at a time until a slight permanent precipitate is formed. This precipitate is dissolved by adding 50 cc of a 4 to 6 per cent solution of sodium potassium tartrate. The sample is now diluted to about 200 cc, solid sodium bicarbonate and starch water added, and the solution titrated with standard iodin.

Total Arsenious Oxid, Method III (Provisional).[2]

SOLUTIONS REQUIRED.

The same solutions as were required for Method II.

METHOD.

Boil 0.4 gram of the finely ground green with 25 cc of sodium acetate (containing 12.5 grams of the crystallized salt) for 5 to 10 minutes. Add concentrated

[1] Has been once recommended by a referee as "optional official." This method can also be used to determine soluble arsenious oxid.

[2] Has been recommended once by a referee as an "optional official" method.

hydrochloric acid a drop at a time until solution of the green is effected (about 10 cc of the acid will be necessary). Add concentrated sodium carbonate solution a drop at a time until a slight precipitate appears, then add 50 cc of a 4 to 6 per cent sodium potassium tartrate solution, and finally sodium bicarbonate in excess. Titrate with standard iodin solution in the usual manner.

Sodium-acetate-soluble Arsenious Oxid (Provisional).

SOLUTIONS REQUIRED.

A solution of sodium acetate containing 12.5 grams of the crystallized salt in each 25 cc; a solution of starch; and a twentieth-normal solution of iodin prepared as indicated above.

METHOD.

Digest 1 gram of paris green over the flame for about 5 minutes, with 25 cc of the sodium acetate solution described above, cool the solution, make up to 100 cc, filter off 50 cc through a dry filter, and titrate with twentieth-normal iodin solution.

Water-soluble Arsenious Oxid (Provisional).

SOLUTIONS REQUIRED.

A starch solution and a twentieth-normal iodin solution, prepared as described above.

METHOD.

One gram of paris green is treated with 1,000 cc of water (previously boiled to expel carbon dioxid, and again cooled to room temperature) in a large flask. The flask is stoppered and shaken eight times each day for 10 days. At the end of this time the solution is filtered off through a dry filter. Two hundred cubic centimeters of this filtrate are treated with sodium bicarbonate and titrated with twentieth-normal iodin solution.

Total Copper Oxid, Method I (Official).

METHOD.

The cuprous oxid obtained in Method I for total arsenious oxid, by boiling the paris green with sodium hydroxid, is poured on the filter and well washed with hot water, after an aliquot portion of the filtrate has been taken for the determination of arsenious oxid. It is then dissolved in hot dilute nitric acid and made up to a volume of 250 cc. Fifty to 100 cc of this solution is used for the electrolytic determination of copper, as described on page 7, paragraph 2-b of Bulletin 46 (revised), Bureau of Chemistry, U. S. Department of Agriculture.

Total Copper Oxid, Method II (Provisional).[1]

SOLUTIONS REQUIRED.

A standard twentieth-normal solution of sodium thiosulphate is prepared by dissolving 24.8 grams of the crystallized salt in 2 liters of water. This solution is standardized against C. P. copper foil dissolved in nitric acid by the method of analysis given in the following paragraph.

METHOD.

An aliquot portion of the nitric acid solution of copper oxid, used in Method I for total copper oxid, is made alkaline with sodium carbonate, then made *slightly* acid with acetic acid, diluted with water, and a considerable quantity

[1] Has been recommended by a referee as an "optional official" method.

of solid potassium iodid added, about 2 or 3 grams. When it is all dissolved by shaking, the free iodin is titrated with the twentieth-normal thiosulphate, using starch as indicator.

Total Copper Oxid, Method III (Provisional).

SOLUTIONS REQUIRED.

A fifth-normal solution of potassium cyanid is prepared, which is standardized against a known weight of copper dissolved in nitric acid, the method being the same as that described in the following paragraph.

METHOD.

An aliquot portion of the nitric acid solution used in Method I for total copper oxid is neutralized with sodium carbonate and a trifling excess of the carbonate added. One cubic centimeter of 0.960 sp. gr. ammonia is added and the dark blue solution titrated to the disappearance of the blue color with standard potassium cyanid.

LONDON PURPLE.

Moisture (Provisional).

Dry from 1 to 2 grams of substance for 10 to 12 hours at a temperature of 105° to 110° C.

Total Arsenious Oxid (Provisional).

SOLUTIONS REQUIRED.

A starch solution and a twentieth-normal iodin solution, prepared by either of the methods given under "paris green."

METHOD.

Two grams of london purple are dissolved in about 80 cc of water and 20 cc of hydrochloric acid at a temperature of 60° to 70° C, filtered, and washed to a volume of 300 cc. One hundred cubic centimeters of this solution are treated with sodium bicarbonate in excess and made up to the mark in a 500-cc flask, using a few drops of ether to destroy bubbles. Two hundred and fifty cubic centimeters are then filtered off through a dry filter, starch water added, and the solution titrated with standard iodin to the appearance of a blue color. The result is the arsenious oxid, as such, in 50 cc of the original solution or 0.3333 gram of the original london purple.

Total Arsenic Oxid, Method I (Provisional).

SOLUTIONS REQUIRED.

The same solutions as those described above for total arsenious oxid are used.

METHOD.

Fifty cubic centimeters of the hydrochloric acid solution of london purple, prepared by the preceding method, is heated to 80° C on the water bath and then taken off and 50 cc of concentrated hydrochloric acid and 3 grams of potassium iodid added. The mixture is allowed to stand for at least 15 minutes, the "ic" arsenic thus being reduced to "ous" arsenic and the iodin set free. The solution is then rinsed out into a large beaker, well diluted, and tenth-normal sodium thiosulphate added drop by drop, to eliminate the free iodin. The end point here is rather difficult to read on account of the very dark color of the solution, but with a little practice the chemist can determine it by proceeding as follows:

The sodium thiosulphate is run in a little at a time, and occasionally a drop of the solution is withdrawn and added to a drop of starch paste. This will, of course, give a blue color with the starch, which becomes fainter as the iodin is used up. Finally, when a drop of the solution gives only the slightest blue color with the starch, a little starch paste is added directly to the whole solution and the blue color dissipated with a few drops of thiosulphate. With a little practice the chemist can in this way get the exact end point. The solution is immediately made alkaline with solid sodium carbonate. It is again made slightly acid with hydrochloric acid, taking care that all of the solid particles of the sodium carbonate on the bottom are neutralized by the acid, and finally made alkaline with sodium bicarbonate. Starch paste is now added and twentieth-normal iodin until the blue color appears. This end point is easily read if the beaker is placed on a white surface between the eye and the light and the iodin solution run in until a distinct purple color appears. The figure thus obtained gives the number of cubic centimeters of iodin corresponding to the total amount of arsenic in the solution as arsenious oxid. Subtracting from this the number of cubic centimeters of iodin corresponding to the arsenious oxid in the previous method gives the number of cubic centimeters of iodin corresponding to the arsenic oxid in 0.3333 gram of the sample.

Total Arsenic Oxid, Method II (Provisional).

(Designed to eliminate part of coloring matter.)

SOLUTIONS REQUIRED.

The same as in preceding method.

METHOD.

One hundred cubic centimeters of the hydrochloric acid solution of london purple prepared under the determination of total arsenious oxid is placed in a beaker and made alkaline with a saturated solution of sodium carbonate. It is then transferred to a 200 cc flask, filled to the mark, and 100 cc filtered off through a dry filter for analysis. This is evaporated to about 50 cc on a hot plate, allowed to cool to about 80° C and 50 cc concentrated hydrochloric acid added, followed by 3 grams of potassium iodid. The mixture is allowed to stand for 15 minutes, the "ic" arsenic being reduced to "ous" arsenic, and the iodin set free. The solution is then rinsed out into a large beaker and the iodin exactly used up with tenth-normal sodium thiosulphate. To get the exact end point it may sometimes be necessary to proceed as in the previous method, but usually there is no trouble. The solution is immediately made alkaline with solid sodium carbonate, again made slightly acid with hydrochloric acid, taking care that all solid particles of sodium carbonate are acted on, and finally made alkaline with sodium bicarbonate. Starch paste is now added and the solution titrated to a blue color. The calculation is the same as that described under Method I for total arsenious oxid.

Soluble Arsenious Oxid (Provisional).

SOLUTIONS REQUIRED.

The same as those used in the preceding method.

METHOD.

Extract 1 gram of london purple in a stoppered flask with 500 cc of cold carbon-dioxid-free water for 7 days, shaking eight times each day. Filter off 100 cc of the solution through a dry filter, add sodium bicarbonate, and titrate with standard iodin, using starch as indicator.

Soluble Arsenic Oxid (Provisional).

SOLUTIONS REQUIRED.

The same solutions as those used in the preceding method.

METHOD.

To determine the soluble arsenic oxid, an aliquot portion of the water extract from the determination of soluble arsenious oxid above (about 200 cc) is transferred to a flask, made slightly alkaline with sodium hydroxid, and evaporated to about 25 cc on a hot plate. The flask is then removed and allowed to cool to about 80° C and an equal volume of concentrated hydrochloric acid and 3 grams of potassium iodid added. It is allowed to stand 15 minutes, the iodin set free is exactly used up with tenth-normal thiosulphate (using starch if necessary), and the solution neutralized with sodium carbonate. It is again made slightly acid with hydrochloric acid, taking care that all lumps of sodium carbonate are acted on, then made alkaline with an excess of sodium bicarbonate, and titrated with iodin, using starch as indicator. From this figure is subtracted the figure representing the amount of soluble arsenious oxid, and the remainder is calculated as arsenic oxid.

COPPER CARBONATE.

Copper Oxid.

SOLUTIONS REQUIRED.

The same solutions as those described under the determination of total copper in paris green are used.

METHODS.

A weighed quantity of the substance is dissolved in nitric acid and the same method of analysis employed as given under the determination of total copper in paris green, Method I being official and Methods II and III provisional.

POTASSIUM CYANID.

Cyanogen (Official).

SOLUTION REQUIRED.

A twentieth-normal solution of silver nitrate.

METHOD.

A large quantity of the sample is weighed out in a weighing bottle, dissolved in water, and made up to a definite volume. Aliquot portions of this are taken for analysis. The twentieth-normal silver nitrate solution is added a drop at a time with constant stirring, until one drop produces a permanent turbidity. In calculating the results, one equivalent of silver is equal to two equivalents of cyanogen, according to the following equation:

$$2\,KCN + AgNO_3 = KCN.AgCN + KNO_3.$$

Cyanogen and Chlorin.

SOLUTIONS REQUIRED.

Same as preceding method.

METHOD.

The cyanogen is determined as in the preceding method. After this is done a few cubic centimeters of a dilute potassium chromate solution are added and the

titration with twentieth-normal silver nitrate continued to the appearance of the red-brown color of silver chromate. The first titration with silver nitrate represents the cyanogen present according to the equation above. The second titration, using potassium chromate as indicator, represents the cyanogen according to the following equation:

$$KCN.AgCN + AgNO_3 = KNO_3 + 2\ AgCN,$$

plus the chlorin present; therefore the second minus the first reading represents the chlorin present in terms of silver nitrate.

SOAP.

General Statement.

In most soaps used by the entomologist (not considering the resin soaps) it is necessary to know three constituents in order to judge of the value of the soap for spraying purposes, viz, moisture, total fatty matter, and total soda or potash. The moisture and alkali are usually determined and the total fatty matter approximately estimated by difference.

Moisture (Provisional).

The following method is given by Benedikt and Lewkowitsch for determining water in soap. Tare accurately a beaker of 100 cc capacity, the bottom of which is covered with recently ignited, perfectly dry sand about one-half inch deep, together with a small glass rod.

Weigh off in a beaker about 5 grams of the sample; add 25 cc of alcohol or more if necessary and dissolve the soap in the alcohol by constant stirring on the water bath. Evaporate the alcohol and finally dry in an oven at 110° C until the weight is constant. A few precautions should be taken which are not mentioned in the above method, viz: If the soap is hard the 5 grams should be cut off in very thin strips so that it will dissolve more readily in the alcohol; also no samples of soap examined by the writer have ever come to a constant weight on drying, but finally begin either to gain or to lose a little indefinitely. It is, therefore, best to heat the soap at 110° C until it is nearly dry and weigh, then return the soap to the oven and dry another half hour. Continue this alternate drying and weighing until the weight changes only a few milligrams during the course of a half hour's drying.

Total Alkali (Provisional).

A weighed quantity of the soap is decomposed by hydrochloric acid and the water is filtered off from the fat, which is washed. Both potassium and sodium in the filtrate are first determined as mixed chlorids in the ordinary manner and the potassium is then determined by means of platinum chlorid.

A rapid but only approximate determination of the alkali in soap is made in the following manner: Weigh out a small quantity of the soap, treat with concentrated sulphuric acid, burn, repeat treatment with sulphuric acid, and burn again. Add a small amount of ammonium carbonate to the disk, cover and heat. Repeat this a number of times till all bisulphates have changed to sulphates. Test the residue qualitatively to determine whether it is sodium or potassium sulphate, and calculate the residue to soda or potash as the case may be.

If the determination of other constituents of the soap be desired the methods given on page 32 of Bulletin 68, Bureau of Chemistry, U. S. Department of Agriculture, have been found fairly satisfactory.

SODA LYE.

Carbonate and Hydroxid, Method I (Provisional).

SOLUTIONS REQUIRED.

A half-normal solution of hydrochloric acid; methyl orange and phenolphthalein indicators.

METHOD.

A large quantity of the sample is weighed out in a weighing bottle, dissolved in water—previously boiled to get rid of carbon dioxid—and made up to a definite volume. Aliquot portions of this are taken for analysis. One portion is titrated with half-normal acid, using methyl orange as indicator, and the total alkalinity thus found noted. Another aliquot portion of the same size is transferred to a measuring flask, and enough barium chlorid added to precipitate out all carbonate, avoiding any unnecessary excess. The volume is made up to the mark with carbon-dioxid-free water, stoppered, shaken, and set aside to allow the precipitate to settle. When the liquid becomes clear one-third of it is drawn off by means of a pipette and titrated with half-normal hydrochloric acid, using phenolphthalein as indicator. This number of cubic centimeters of half-normal acid multiplied by 3 gives the number of cubic centimeters of half-normal acid corresponding to the original amount taken. The last figure obtained represents sodium hydroxid, and the difference between the first and last figures represents the sodium carbonate.

Carbonate and Hydroxid, Method II (Provisional).

SOLUTIONS REQUIRED.

A fifth-normal solution of potassium acid sulphate; methyl orange and phenolphthalein indicators.

METHOD.

The lye is weighed out and dissolved to a definite volume in carbon-dioxid-free water just as described in Method I. An aliquot portion is taken, diluted with carbon-dioxid-free water and a few drops of phenolphthalein added. A fifth-normal solution of potassium acid sulphate is added at the rate of about 1 drop per second, with constant stirring, until the pink color fades out and the solution becomes colorless. The reading thus obtained, which we will call n, represents the sodium hydroxid and one-half of the sodium carbonate present since the sodium carbonate is changed to sodium bicarbonate. Methyl orange is added and the titration continued to the appearance of a pink color. Let this number of cubic centimeters be represented as m. Since this last titration represents the sodium bicarbonate present, or one-half the sodium carbonate, $2m$ represents all the sodium carbonate present and $n - m$ the sodium hydroxid.

TOBACCO AND TOBACCO EXTRACTS.

Nicotin, Method I (Official).

SOLUTIONS REQUIRED.

An alcoholic soda solution containing 6 grams of sodium hydroxid, 40 cc of water, and 60 cc of 90 per cent alcohol. A weak sodium hydroxid solution containing 4 grams of sodium hydroxid in 1,000 cc of water. A standard sulphuric acid solution.

METHOD.

About 5 to 6 grams of tobacco extract or 20 grams of finely powdered tobacco, which has been previously dried at 60° C so as to allow it to be powdered, is weighed out into a small beaker. Ten cubic centimeters of the alcoholic soda solution is added, followed, in the case of the tobacco extract, with enough C. P. powdered calcium carbonate to form a moist but not lumpy mass. The whole is well mixed. This is transferred to a Soxhlet extractor and exhausted for about 5 hours with ether. The ether is evaporated off at a low temperature by being held over the steam bath, and the residue is taken up with 50 cc of the weak soda solution mentioned above under "solutions required." This is transferred by means of water to a Kjeldahl flask, capable of holding about 500 cc, and distilled in a current of steam, using a condenser through which water is flowing rapidly. A three-bend outflow tube is used, and a few pieces of pumice and a small piece of paraffin are added to prevent bumping and frothing. The distillation is continued till all the nicotin has passed over, the distillate usually varying from 400 to 500 cc. When the distillation is complete only about 15 cc of the liquid should remain in the distillation flask. The distillate is titrated with standard sulphuric acid, using phenacetolin or cochineal as indicator. One molecule of sulphuric acid is equivalent to two molecules of nicotin.

Nicotin, Method II (Provisional).

SOLUTIONS REQUIRED.

A weak sodium hydroxid solution containing 4 grams to the liter and a standard fifth-normal hydrochloric acid solution.

METHOD.

A solution of C. P. nicotin of approximately 1 per cent strength is first prepared and an aliquot portion carefully titrated against fifth-normal hydrochloric acid, using methyl orange as indicator, so as to definitely establish the exact percentage of nicotin in solution. A separate portion of the same solution is then placed in a 40-cm tube and its rotation in terms of the sugar scale determined. By dividing the percentage of nicotin present by the reading on the sugar scale the value of one degree of the scale in terms of per cent of nicotin is established and the figure so obtained can be used as a factor in all determinations of nicotin made, provided the same polariscope is used.

A weighed amount of the sample, about 3 to 5 grams, is now rendered alkaline with 50 cc of the weak soda solution, mentioned under "solutions required," and transferred to a round-bottomed flask of about 300 to 500 cc capacity with 150 cc of distilled water. The whole is now subjected to distillation in a current of steam and the distillate made to a volume of 500 cc. A portion of the distillate is then placed in a 40-cm tube and a polariscope reading taken. Knowing the value of each degree of the sugar scale in terms of per cent of nicotin in the solution, the per cent of nicotin in the sample can be calculated.

FORMALIN.

Formaldehyde, Method I.

SOLUTIONS REQUIRED.

A normal solution of sulphuric acid, a normal solution of sodium hydroxid, and a solution of purified litmus.

METHOD.

Measure out 50 cc of normal sodium hydroxid in a small Erlenmeyer flask (of 500 cc capacity, for example) and add 50 cc of hydrogen dioxid. Now add 3 cc of the formaldehyde solution under examination (the specific gravity of which has been previously determined), allowing the point of the pipette to almost reach the liquid in the flask. Place a funnel in the neck of the flask and put on the steam bath for 5 minutes, shaking occasionally during this time. Remove from the steam bath, wash the funnel with distilled water, cool the flask down to about room temperature, and titrate the excess of sodium hydroxid with normal acid, using litmus as indicator. This cooling of the flask before titration with acid was found necessary in order to get a sharp end reading with the litmus. From the volume of formaldehyde used and the specific gravity the per cent of formaldehyde can be determined.

Formaldehyde, Method II (Provisional).[1]

SOLUTIONS REQUIRED.

A tenth-normal solution of silver nitrate, a tenth-normal solution of ammonium sulpho-cyanate, a dilute solution of potassium cyanid containing 3.1 grams to 500 cc of water, and a 50 per cent solution of nitric acid.

METHOD.

Fifteen cubic centimeters of tenth-normal silver nitrate are treated with 6 drops of 50 per cent nitric acid in a 50-cc flask; 10 cc of a solution of potassium cyanid (containing 3.1 grams of potassium cyanid in 500 cc of water) are then added and well shaken. The solution is then made to the mark and an aliquot portion of the filtrate (say 25 cc) is titrated according to the method of Volhard with a tenth-normal solution of ammonium sulpho-cyanate for the excess of silver. Another 15-cc portion of tenth-normal silver nitrate is acidified with 6 drops of 50 per cent nitric acid and treated with 10 cc of the potassium cyanid solution to which has been added a weighed quantity of the dilute formaldehyde solution. The whole is made up to 50 cc and a 25-cc filtrate from it is titrated with tenth-normal ammonium sulpho-cyanate for the excess of silver as before. The difference between these results multiplied by 2 gives the amount of potassium cyanid that has been used by the formaldehyde in terms of tenth-normal ammonium sulpho-cyanate. Each cubic centimeter of this is equivalent to 3 milligrams of formaldehyde.

Lime-sulphur dips and lime-sulphur-salt mixture (provisional).

Total Sulphur.

SOLUTIONS REQUIRED.

A saturated potassium hydroxid solution or a solution of caustic soda containing 100 grams to 100 cc of water. A 10 per cent barium chlorid solution. An approximately 3 per cent solution of hydrogen peroxid free from sulphates; if it contains sulphates add freshly precipitated barium carbonate and shake occasionally for several hours, then filter and use the clear solution.

METHOD.

Measure off 10 cc of the clear sample in a 100-cc measuring flask and fill to the mark. Use aliquot portions of 10 cc of this for analysis. Treat this aliquot

[1] Has been recommended once as an official method.

with 3 cc of the caustic potash or soda solution, following this by 50 cc of hydrogen peroxid free of sulphates. Heat on the steam bath for just one-half hour and then acidify with hydrochloric acid, precipitate with barium chlorid in the ordinary manner in boiling solution, and finally weigh as barium sulphate.

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J. K. Haywood,
Chief, Miscellaneous Laboratory.

Approved:
James Wilson,
Secretary.

Washington, D. C., *August 21, 1905.*

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